भारतीय मानक
Indian Standard

IS 15081 : 2022

एन,एन–बिसहाइड्रोक्सीइथायिल-मेटा-क्लोरोएनिलीन, तकनीकी — विशिष्टि

(*पहला पुनरीक्षण*)

N,N-Bishydroxyethyl-Meta-Chloroaniline, Technical — Specification

(*First Revision*)

ICS 71.080.30

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भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
मानक भवन, 9 बहादुर शाह ज़फर मार्ग, नई दिल्ली - 110002
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI - 110 002
www.bis.gov.in www.standardsbis.in

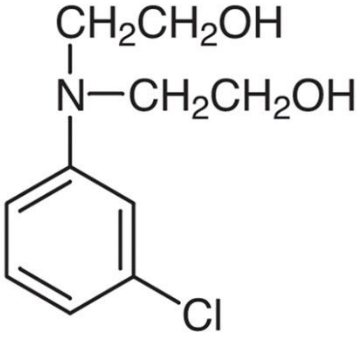
October 2022 **Price Group 8**

Dye Intermediates Sectional Committee, PCD 26

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

N,N-Bishydroxyethyl-meta-chloroaniline ($C_{10}H_{14}O_2NCl$) is an important dye intermediate used in the manufacture of azo disperse dyes. It has the following structural formula:



N,N-BISHYDROXYETHYL-META-CHLOROANILINE

[MOLECULAR MASS: 215.5]

(CAS REGISTRY NO.:92-00-2)

This Standard was first published in 2001. The Committee responsible for the preparation of this standard decided to revise it in order to incorporate recent development in analytical techniques and use of more sophisticated instrument to determine purity and impurity profile.

In this (first) revision, test methods for determination of purity by gas chromatography (GC) and impurity of meta-chloroaniline and N-hydroxyethyl-meta-chloroaniline content have been updated. In addition to these, requirement for chlorobenzene's (CB's) and chlorotoluene's (CT's) are also incorporated considering to environmental hazard of these derivatives.

The composition of the Committee responsible for formulation of this standard is given in Annex D.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

N,N-BISHYDROXYETHYL-META-CHLOROANILINE,TECHNICAL — SPECIFICATION

(*First Revision*)

1 SCOPE

This standard prescribes the requirements, methods of sampling and testing for N,N-bishydroxyethyl-meta-chloroaniline.

2 REFERENCES

The following standards contain provisions, which through reference in this text constitute provisions of this standards. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
1070 : 1992	Reagent grade water — Specification (*third revision*)
2552 : 1989	Steel drums (galvanized and ungalvanized) — Specification (*third revision*)
5299 : 2001	Methods of sampling and tests for dye intermediates (*first revision*)

3 REQUIREMENTS

3.1 Description

The material shall be in the form of cream solid material.

3.2 The material shall also comply with the requirements given in Table 1, when tested according to the methods prescribed in col 4 and col 5 of Table 1.

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in steel drums (*conforming to* IS 2552) lined with suitable polyethylene film or as agreed to between the purchaser and the supplier. Each container shall be securely closed.

4.2 Marking

4.2.1 Each container shall be securely closed and shall bear legibly and indelibly the following information:

a) Name of the material;
b) Name of manufacturer and his recognized trade-mark, if any;
c) Net mass of material;
d) Month and year of the manufacture;
e) Batch or lot number; and
f) Any other statutory requirements.

Table 1 Requirements for N,N-Bishydroxyethyl-meta-chloroaniline, Technical

(*Clauses* 3.2, 5.3.1, 5.3.2 *and* 6.1)

Sl No.	Characteristics	Requirement	Method of Test, Ref to	
			Annex	Clause No. in IS 5299
(1)	(2)	(3)	(4)	(5)
i)	Melting Range, °C	87-90	—	**9**
ii)	Purity by titration method, percent by mass, *Min*	97.00	A	—
	Or			
	Purity by GC$^{1)}$,percent by mass, *Min*	97.00	B	—
iii)	N-hydroxyethyl-meta-chloroaniline, *Max*	1.5	B	—
iv)	Meta-chloroaniline (on dry basis), *Max*	0.2	B	—
v)	Insoluble in methanol, percent by mass, *Max*	0.3	—	**11**
vi)	Sum of chlorobenzene’s and chlorotoluene’s, ppm, *Max*	50	C	—

$^{1)}$ In case of disputes, purity by GC shall be the referee method.

4.2.2 *BIS Certification Marking*

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act,* 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

5 SAMPLING

5.1 The method of drawing representative samples of the material shall be as prescribed in **4** of IS 5299.

5.2 Numbers of Tests

5.2.1 Tests for the determination of purity, meta-chloroaniline and N-hydroxyethyl-meta-chloroaniline content shall be conducted on the individual samples.

5.2.2 Tests for the determination of remaining characteristics, namely, description, melting range, insoluble in methanol and sum of chlorobenzene’s and chlorotoluene’s shall be conducted on the composite sample.

5.3 Criteria for Conformity

5.3.1 *For Individual Samples*

The lot shall be declared as conforming to the requirement of N,N-bishydroxyethyl-meta-chloroaniline, meta-chloroaniline and N-hydroxyethyl-meta-chloroaniline, if each of the individual test results satisfies the relevant requirements given in Table 1.

5.3.2 *For Composite Sample*

For declaring the conformity of the lot to the requirements of all other characteristics (**5.2.2**) tested on the composite sample, the test results for each of the characteristics shall satisfy the relevant requirements given in **3.1** and Table 1.

6 TEST METHODS

6.1 Tests shall be carried out according to the methods referred in col 4 and col 5 of Table 1.

6.2 Quality of Reagents

Unless specified otherwise, 'pure chemicals' and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

[*Table* 1, *Sl No.*(ii)]

DETERMINATION OF N,N-BISHYDROXYETHYL-META-CHLOROANILINE CONTENT (PURITY) BY TITRATION METHOD

A-1 OUTLINE OF THE METHOD

A diazonium solution is prepared by reacting 2,4-dinitroaniline in concentrated sulphuric acid with sodium nitrite solution. It is standardized by titrating against N,N-bishydroxyethyl-meta-chloroaniline of known purity. The sample of N,N-bishydroxyethyl-meta-chloroaniline is then titrated against this standard diazonium solution.

A-2 REAGENTS

A-2.1 Glacial Acetic acid

A-2.2 Dilute Sulphuric Acid, 20 percent (*m/v*).

A-2.3 Dilute Acetic Acid, 20 percent (*m/v*).

A-2.4 Sodium Acetate Solution, 20 percent (*m/v*).

A-2.5 H-Acid Indicator Solution

Dissolve 0.5 g of H-acid in 100 ml of water containing 1 g of soda ash.

A-2.6 2,4-Dinitroaniline, 99 percent pure.

A-2.7 N,N-Bishydroxyethyl-meta-Chloroaniline, reference sample of known purity.

A-3 PROCEDURE

A-3.1 Preparation of 2,4-Dinitroaniline Diazo Solution

Take 600 g (326 ml) of concentrated sulphuric acid (H_2SO_4) in a reaction vessel with stirrer which is cooled to 10 °C externally on ice bath. Add 69 g of sodium nitrite (100 percent) gradually under stirring maintaining the temperature below 10 °C. After the addition is complete, remove the ice bath and bring it to room temperature and then raise to 60 °C to dissolve the material completely. After the dissolution is complete the temperature is again brought below 20 °C. Similarly, prepare a slurry of 2,4-dinitroaniline in sulphuric acid (H_2SO_4), by dissolving 183 g of 2,4-dinitroaniline in 200 g of sulphuric acid (H_2SO_4) in a 500 ml beaker. Add this slurry to the nitrosulphuric acid slurry prepared earlier maintaining the temperature below 20 °C. After the addition is complete, stir for another 2 h for the completion of reaction (stock solution). Depending upon the requirements, a portion of this slurry is taken out and diluted with 10 times by its volume with ice water (for example, 10 ml of stock solution and 90 ml of ice water). The solution is then clarified with activated carbon and filtered before use. It is to be noted that the diluted diazo solution is not stable and hence should be prepared fresh every time before use.

A-3.2 Standardization of 2,4-Dinitroaniline Diazonium Solution

Weigh exactly 0.780 8 g of N,N-bishydroxyethyl-meta-chloroaniline (99 percent) and transfer it to a 1 l beaker. Dissolve it with 10-15 ml of glacial acetic acid and dilute the solution with ice water to 500 ml. Add about 100 ml of 20 percent sodium acetate solution (*m/v*) to bring the *p*H between 4 to 6. Titrate with 2,4-dinitroaniline diazonium (diluted) until the solution shows the presence of an excess diazonium with H-acid (bluish black coloration). This may be checked by taking a drop of the reaction mixture and a drop of a diluted solution of H-acid on a piece of filter paper. The bluish black coloration in the junction of the two liquids indicates that the end point has reached. Throughout the titration, the *p*H should be maintained between 4 to 6.

A-3.3 Repeat the titration as presented in **A-3.2** using the test sample of N,N-bishydroxyethyl-meta-chloroaniline.

A-4 CALCULATION

A-4.1 Normality of diazonium solution

$$N = \frac{P \times 0.780}{V \times 21.55}$$

where

P = purity of the known sample, percent by mass; and
V= volume of diazonium solution required for the known sample, ml.

A-4.2 Purity, percent by mass

$$= \frac{V_1 \times N \times 21.55}{0.780}$$

where

V_1 = volume of diazonium solution required for the test sample, ml; and
N = normality of diazonium solution.

ANNEX B

[*Table* 1, *Sl No.*(ii),(iii) and (iv)]

DETERMINATION OF N,N-BISHYDROXYETHYL-META CHLOROANILINE CONTENT (PURITY) BY GAS CHROMATOGRAPHY

B-1 OUTLINE OF THE METHOD

The contents of N,N-bishydroxyethyl-meta-chloroaniline and possible impurities, namely, N-hydroxyethyl-meta-chloroaniline and meta-chloroaniline are determined by gas liquid chromatographic method.

B-2 APPARATUS

B-2.1 Any gas chromatograph equipped with a flame ionization detector (FID), a split/splitless injector and a suitable electronic integrator/software can be used can be used with following accessories and operating condition:

B-2.1.1 Column, 5 percent phenyl, 95 percent methylpolysiloxane, bonded and cross-linked phase with length 15 m, internal diameter 0.25 mm and film thickness 1.50 µm or equivalent.

B-2.1.2 GC Parameters

Injector	Split Temperature: 260°C Split ratio: 10
Carrier gas	Nitrogen or Helium with flow control mode pressure
Injection volume	1.0 µl

B-2.1.3 FID Parameters

Hydrogen flow	40 ml/min
Air flow	400 ml/min
Temperature	290 °C

B-2.1.4 Oven Temperature

Heating rate, °C/min	*Temperature, °C*	*Time, min*
—	100	0
10	290	10

NOTE — The above gas chromatographic (GC) conditions are suggestive. However, any GC method having difference in detector, column packing material and type (like packed/capillary, diameter, length, film thickness,etc), calibration technique (internal standard, external standard, area normalization, percent area, etc), carrier gas (He, H_2, N_2) may be used with applicable GC operating parameters, provided standardization and calibration of the components is established after setting GC parameters for the resolution and accuracy level as specified in this standard.

B-3 REAGENT

B-3.1 Acetone, AR grade.

B-3.2 N,N-Bishydroxyethyl-meta-chloroaniline

B-3.3 *m*-Chloroaniline

B-3.4 N-Hydroxyethyl-meta-chloroaniline

B-4 STANDARD PREPARATION

Weight accurately 1 g of standard N,N-bishydroxyethyl-meta-chloroaniline in 100 ml volumetric flask. Dissolve it in acetone and make the volume up to the mark with acetone.

B-5 SAMPLE PREPARATION

Weight accurately 1 g of sample in 100 ml volumetric flask dissolve in acetone and make up the volume up to the mark with acetone.

B-6 RETENTION TIME

Meta-chloroaniline	4.53 min
N-Hydroxyethyl-meta-chloroaniline (Mono)	9.74 min
N,N-Bishydroxyethyl-meta-chloroaniline	13.74 min

B-7 CALCULATION

Calculate the peak area of individual constituent pertaining to N,N-bishydroxyethyl-meta-chloroaniline on the chromatogram of the material. The concentration of the constituent may be obtained on the basis of peak area on chromatogram obtained with standard N,N-bishydroxyethyl-meta-chloroaniline.

$$\mathrm{N,N-bishydroxyethyl-meta-chloroaniline, percent} = \frac{A_2 \times V_1 \times W_1 \times B_2}{A_1 \times V_2 \times W_2 \times B_1} \times 100$$

where

A_1 = area of standard N,N-bishydroxyethyl-meta-chloroaniline;

V_1 = injection volume of standard N,N-bishydroxyethyl-meta-chloroaniline;

W_1 = weight of standard N,N-bishydroxyethyl-meta-chloroaniline;

B_1 = total volume of standard N,N-bishydroxyethyl-meta-chloroaniline;

A_2 = area of N,N-bishydroxyethyl-meta-chloroanilinepeak in sample;

V_2 = injection volume of sample; and

W_2 = weight of sample.

B_2 = total volume of sample.

ANNEX C

[*Table* 1, *Sl No.*(vi)]

DETERMINATION OF CHLOROBENZENE'S (CB's)ANDCHLOROTOLUENE'S (CT's) BY GAS CHROMATOGRAPHY MASS SPECTROMETER

C-1 APPARATUS

C-1.1 Any gas chromatograph equipped with a mass spectroscopy detector, a split/splitless injector and a suitable electronic integrator/software can be used can be used with following accessories and operating condition:

C-1.1.1 Column, 65 percent dimethylpolysiloxane and 35 percent phenyl phase with length 30 m, internal diameter 0.25 mm, film thickness 0.25 µm and column maximum temperature 340°C or equivalent.

C-2 INJECTION PORT INFORMATION

Injection Mode	*Temperature*	*Column Flow*	*Purge Flow*	*Split Ratio*	*Injection Volume*
Split	240 °C	1.00 ml/min	3.0 ml/min	2.0	1 µl

C-3 COLUMN OVEN INFORMATION

Initial Temperature	*Equilibration time*	*Total Program Time*
40 °C	3.00	36 min

C-4 COLUMN OVEN TEMPERATURE PROGRAM

Rate, °C/min	*Temperature, °C*	*Hold Time, min*
—	40	0.5
5	140	1
20	290	6

C-5 DETECTOR INFORMATION

Detector	*Iron Source Temperature*	*Interface Temperature*	*Solvent Cut Time*	*Detector Voltage*	*Threshold*
MS	230 °C	280 °C	3 min	Relative + 0.3 kV	100

Start Time	*End Time*	*Acquire mode*	*M/Z*
3	11	SIM	63,77,91,112,114,126
11	15	SIM	65,91,111,114,146,148
15	20	SIM	125,127,145,160,180,182
20	26.5	SIM	159,161,194,196,214,218,240,250,252
26.5	29.5	SIM	229,284,286

C-6 AUTO INJECTION INFORMATION

Rinses with Solvent (pre Run)	*Rinses with Solvent (post Run)*	*Rinses with Sample*	*Plunger speed*	*Viscosity Comp. temp*	*Plunger Speed*	*Syringe inserting Speed*
0	5	2	High	0.2 s	High	High

C-7 REAGENTS

C-7.1 Monochlorobenzene[MCB]

C-7.2 1,2-dichlorobenzene [ODCB]

C-7.3 1,3-dichlorobenzene [MDCB]

C-7.4 1,4-dichlorobenzene [PDCB]

C-7.5 1,2,3-trichlorobenzene [1,2,3-TCB]

C-7.6 1,2,4-trichlorobenzene [1,2,4-TCB]

C-7.7 1,3,5-trichlorobenzene [1,3,5-TCB]

C-7.8 1,2,3,4-tetrachlorobenzene [1,2,3,4-TeCB]

C-7.9 1,2,3,5-tetrachlorobenzene [1,2,3,5-TeCB]

C-7.10 1,2,4,5-tetrachlorobenzene [1,2,4,5-TeCB]

C-7.11 Pentachlorobenzene [PCB]

C-7.12 Hexachlorobenzene [HCB]

C-7.13 Toluene

C-7.14 2-chlorotoluene [2-CT]

C-7.15 3-chlorotoluene [3-CT]

C-7.16 4-chlorotoluene [4-CT]

C-7.17 2,3-dichlorotoluene [2,3-DCT]

C-7.18 2,4-dichlorotoluene [2,4-DCT]

C-7.19 2,5-dichlorotoluene [2,5-DCT]

C-7.20 2,6-dichlorotoluene [2,6-DCT]

C-7.21 3,4-dichlorotoluene [3,4-DCT]

C-7.22 2,3,6-trichlorotoluene [2,3,6-TCT]

C-7.23 2,4,5-trichlorotoluene [2,4,5-TCT]

C-7.24 Pentachlorotoluene [PCT]

C-7.25 Dichloromethane [DCM]

C-7.26 Methanol

C-8 Retention time of the CB’s

Product Name	*Retention Time*	*M/Z*
MCB	6.59	112
MDCB	11.33	146
PDCB	11.68	146
ODCB	12.57	146
1,3,5 TCB	14.97	180
1,2,4 TCB	16.83	180
1,2,3 TCB	18.21	180
1,2,3,5 TeCB	21.04	216
1,2,4,5 TeCB	21.13	216
1,2,3,4 TeCB	22.88	216
Penta CB	24.93	250
Hexa CB	26.75	284

Product Name	*Retention Time*	*M/Z*
Toluene	4.43	91
2-CT	9.52	91
3-CT	9.59	91
4-CT	9.65	91
AlphaCT	12.36	91
2,4-DCT	14.67	125
2,6-DCT	14.96	125
2,5-DCT	14.91	125
3,4-DCT	15.87	125
2,3-DCT	15.97	125
2,4,5-TCT	20.04	159
Penta CT	36.44	229

C-9 STANDARD PREPARATION

C-9.1 Stock Solution

Weigh 0.002 5 g each standard in each 25 ml volumetric flask and fill it up to the mark with methanol.

Stock solution of each standard is 100 ppm.

C-9.2 Stock Solution No.1 except 2,6-DCT and 3,4-DCT

Take 1 ml of each stock solution in 100 ml volumetric flask and fill it up to the mark with DCM (1 ppm each mixture solution).

C-9.3 Stock Solution No.2

Take 10 ml of above stock solution No.1 in 100 ml volumetric flask and fill it up to the mark with DCM (0.1 ppm each mixture solution).

C-9.4 Stock Solution No.3

Take 5 ml of above stock solution No.1 in 100 ml volumetric flask and fill it up to the mark with DCM (0.05 ppm each mixture solution).

C-9.5 Stock Solution No.4

Take 1 ml of above stock solution No.1 in 100 ml volumetric flask and fill it up to the mark with DCM (0.01 ppm each mixture solution).

C-9.6 Stock Solution No.5

Take 10 ml of above stock solution No.3 in 100 ml volumetric flask and fill it up to the mark with DCM (0.01 ppm each mixture solution).

C-10 SAMPLE PREPARATION

Weigh 0.1 g sample into a 10 ml volumetric flask. Add 5 ml DCM and shake it well on cyclo mixer. After 30 min fill it up to the mark with DCM. The solution has to be filtered before injection into the GC-MS.

If necessary dilute the sample solution according to the calibration range.

C-11 CALCULATION

$$\text{Chlorobenzene's (CB's), ppm} = \frac{A_2 \times V_1 \times W_1 \times B_2}{A_1 \times V_2 \times W_2 \times B_1} \text{x } 10^6$$

where

A_1 = area of standard chlorobenzene’s (CB’s);

V_1 = injection volume of standard chlorobenzene’s (CB’s);

W_1 = weight of standard chlorobenzene’s (CB’s);

B_1 = total volume of standard chlorobenzene’s (CB’s);

A_2 = area of chlorobenzene’s (CB’s) peak in sample;

V_2 = injection volume of sample;

W_2 = weight of sample; and

B_2 = total volume of sample.

Similarly, contents of chlorotoluene’s (CT’s) shall be calculated.

C-12 REPORT

Result shall be reported as sum of chlorobenzene’s (CB’s) and chlorotoluene’s (CT’s).

ANNEX D

(*Foreword*)

COMMITTEE COMPOSITION

Dye Intermediates Sectional Committee, PCD 26

Organization	*Representative(s)*
Institute of Chemical Technology, Mumbai	Prof Ganapati Subray Shankarling (***Chairperson***)
Aarti Industries Limited, Mumbai	Dr Vaishali Bhandary Dr Sanjeev Kumar Dixit (*Alternate)*
Alkyl Amines Chemicals Limited, Mumbai	Shri S. V. Nikumbhe Shri Kirat Patel (*Alternate*)
Ankleshwar Research and Analytical Infrastructure Limited, Ankleshwar	Shri Mansukh H Vekaria
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Central Revenue Control Laboratory, New Delhi	Dr T. A. Sreenivasa Rao Shri Praful Dalal (*Alternate*)
Colourtex Industries Limited, Mumbai	Dr Pankaj Desai Shri R. K. Jaiswal (*Alternate*)
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Dystar, Mumbai	Dr Monika Singh
Ecological and Toxicological Association of Dyes, Vadodara	Dr Pariti Siva Rama Kumar
Gujarat Dyestuffs Manufacturers Association, Ahmedabad	Shri Yogesh Parikh Shri Anil M. Jain (*Alternate* I) Shri Haresh Bhuta (*Alternate* II)
Gujarat Narmada Valley Fertilizers Company Limited, Ahmedabad	Shri R. M. Patel Shri C. S. Patel (*Alternate*)
Gujarat Pollution Control Board, Gandhinagar, Ahmedabad	Shri Y. A. Tai

Organization	*Representative(s)*
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Indian Beauty and Hygiene Association, Mumbai	MS MALATHI NARAYANAN
Indian Chemical Council, Mumbai	SHRI P. S. SINGH
Jay Chemicals Industries Private Limited, Ahmedabad	SHRI VILPESH YADAV SHRIMATI MAITRI VYAS (*Alternate*)
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National Test House, Kolkata	SHRI P. K. CHAKRABORTY SHRI Y. C. NIJHAWAN (*Alternate*)
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The Bombay Textile Research Association, Mumbai	MS SHITAL PALASKAR
The Dyestuff Manufactures Association of India Office, Mumbai	SHRI V. R. KANETKAR
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Member Secretary
MS ADITI CHOUDHARY
Scientist 'B', BIS

Working Group for validation of test methods, PCD 26/ WG-1

Organization	*Representative(s)*
Colourtex Industries Limited, Mumbai	DR PANKAJ DESAI (***Convener***)
Aarti Industries Limited, Mumbai	DR VAISHALI BHANDARY
Atul Limited, Gujarat	DR M. U. RAHMAN
Archroma India Private Limited, Thane	DR RAJESH RAMAMURTHY
Central Revenue Control Laboratory, New Delhi	DR T. A.SREENIVASA RAO
Heubach Colour Pvt Ltd, Mumbai	SHRI J. I. SEVAK
Jay Chemicals Industries Pvt Ltd, Ahmedabad	SHRI VILPESH YADAV
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Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones: 2323 0131, 2323 3375, 2323 9402 *Website*: www.bis.gov.in

Regional Offices:		*Telephones*
Central	: 601/A, Konnectus Tower -1, 6th Floor, DMRC Building, Bhavbhuti Marg, New Delhi 110002	2323 7617
Eastern	: 8th Floor, Plot No 7/7 & 7/8, CP Block, Sector V, Salt Lake, Kolkata, West Bengal 700091	2367 0012 2320 9474
Northern	: Plot No. 4-A, Sector 27-B, Madhya Marg, Chandigarh 160019	265 9930
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Western	: Plot No. E-9, Road No.-8, MIDC, Andheri (East), Mumbai 400093	2821 8093

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